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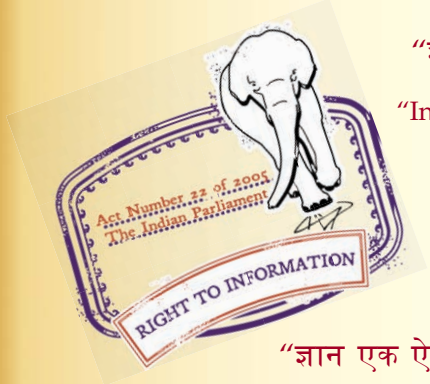
IS 8120 (1989): Schaeffer's Acid (Sodium Salt), Technical [PCD 9: Organic Chemicals Alcohols and Allied Products and Dye Intermediates]



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"Knowledge is such a treasure which cannot be stolen"

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IS 8120 : 1989
(Reaffirmed 2000)

Indian Standard

SCHAEFFER'S ACID (SODIUM SALT), TECHNICAL — SPECIFICATION

(First Revision)

भारतीय मानक

शैफर एसिड (सोडियम लवण), तकनीकी — विशिष्टि

(पहला पुनरीक्षण)

UDC 547·655·2

BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

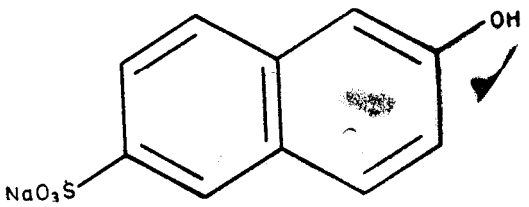
November 1989

Price Group 2

FOREWORD

This Indian Standard (First Revision) was adopted by the Bureau of Indian Standards on 20 February 1989, after the draft finalized by the Dye Intermediates Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

Schaeffer's acid ($C_{10}H_7O_4SNa$) is a dye intermediate. It is chemically known as 2-naphthol-6-sulphonic acid. It is available as its mono sodium salt and is represented by the following structural formula.



SCHAEFFER'S ACID (SODIUM SALT)

(Molecular mass 246)

CAS Registry Number [93-01-6]

This standard was first published in 1976. The Committee responsible for the preparation of the standard decided to revise it in order to update the same in the light of the experience gained over the past few years. In the present version, the requirement of β-naphthol has been made more stringent and the requirement of impurities like R Salt and G Salt along with paper chromatographic method for their estimation has been introduced.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

SCHAEFFER'S ACID (SODIUM SALT), TECHNICAL — SPECIFICATION

(First Revision)

1 SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for Schaeffer's acid (sodium salt), technical.

2 REFERENCES

2.1 The following Indian Standards are necessary adjuncts to this standard:

IS No.	*Title*
IS 1070 : 1977	Specification for water for general laboratory use (*second revision*)
IS 2552 : 1979	Specification for steel drums (galvanized and ungalvanized) (*second revision*)
IS 5299 : 1969	Methods of sampling and tests for dye intermediates

3 REQUIREMENTS

3.1 Description

The material shall be in the form of off-white powder or off-white moist cake.

3.2 The material shall also comply with the requirements given in Table 1.

Table 1 Requirements for Schaeffer's Acid (Sodium Salt), Technical

Sl No.	Characteristic	Requirement	Method of Test, Ref to Clause No. in Annex A
(1)	(2)	(3)	(4)
i)	Assay (on dry basis), percent by mass, *Min*	75	A-2
ii)	β-Naphthol content (on dry basis), percent by mass, *Max*	0·5	A-3
iii)	R salt (on dry basis), percent by mass, *Max*	1·0	A-3
iv)	G salt (on dry basis), percent by mass, *Max*	1·0	A-3
v)	Matter insoluble in sodium carbonate solution (on dry basis), percent by mass, *Max*	0·30	A-4

4 PACKING AND MARKING

4.1 Packing

The material shall be packed in steel drums (*see* IS 2552 : 1979) lined with suitable polyethylene film or as agreed to between the purchaser and the supplier.

4.2 Marking

Each container shall be securely closed and shall bear legibly and indelibly the following information:

a) Name of the material;
b) Name of the manufacturer and his recognized trade-mark, if any;
c) Batch number; and
d) Gross mass, tare and net mass.

5 SAMPLING

5.1 Representative samples of the material shall be drawn as prescribed in **3** of IS 5299 : 1969.

5.2 Number of Tests

5.2.1 Test for assay shall be conducted on each of the individual samples.

5.2.2 Test for determination of remaining characteristics, namely, β-naphthol and solubility and sodium hydroxide solution shall be conducted on the composite sample.

5.3 Criteria for Conformity

5.3.1 *For Individual Samples*

The lot shall be declared as conforming to the requirements of assay if each of the individual test results satisfies the relevant requirement given in Table 1.

5.3.2 *For Composite Sample*

For declaring the conformity of the lot to the requirements of all other characteristics tested on the composite sample (*see* **5.2.2**), the test results for each of the characteristics shall satisfy the relevant requirement given in Table 1.

6 TEST METHODS

6.1 Tests shall be carried out according to the methods prescribed in Annex A as indicated in col 4 of Table 1.

6.2 Quality of Reagents

Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070 : 1977) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

ANNEX A

(*Table* 1)

METHODS OF TEST FOR SCHAEFFER'S ACID (SODIUM SALT), TECHNICAL

A-1 PREPARED SAMPLE

A-1.1 Dry the material at 110 ± 1°C to constant mass. Grind and mix well. Transfer the material to a wide-mouthed bottle and stopper it. Do not expose the sample to an atmosphere containing acidic or alkaline fumes. Use this prepared sample for tests.

A-2 ASSAY

A-2.1 Outline of the Method

The material is dissolved in water adding sodium carbonate. A known volume of the solution is titrated against standard 4-chlorobenzene diazo solution in alkaline medium and, from the consumption of the diazonium compound, strength is calculated

A-2.2 Reagents

A-2.2.1 *Sodium Carbonate Solution*, approximately 10 percent (*m/v*).

A-2.2.2 *Phenolphthalein Indicator Paper*

A-2.2.3 *Standard 4-Chlorobenzene Diazo Solution*, 0·1 N.

A-2.2.4 *H Acid Indicator*

Dissolve 0·5 g of H acid in 100 ml of 1 percent ammonium hydroxide solution.

A-2.3 Procedure

Weigh accurately about 5 g of the prepared sample (*see* **A-1.1**) and transfer to a 500-ml beaker with the help of water. Slowly add sodium carbonate solution to get a positive test on phenolphthalein paper. Transfer the solution quantitatively into a 500-ml volumetric flask and dilute to mark with water. Mix well. Pipette 50 ml aliquot of this solution into a 1-litre beaker. Add 200 ml of ice-cold water. Add 50 ml of sodium carbonate solution. Stir with a glass rod and cool with washed ice to below 1°C. Titrate with 4-chlorobenzene diazonium solution from a cold water-jacketted burette. Test with H acid indicator for the excess of diazo and with the diazo for the coupling component. The end point is noted when there is no test with diazo and a fine visible pink line with H acid indicator is obtained which can persist for 10 minutes without further addition of diazo. Let the titre reading be V.

A-2.4 Calculation

$$\text{Assay, percent by mass} = \frac{V \times N \times 246}{M}$$

where

V = volume in ml of the standard diazonium solution used,

N = mass in g of the material taken for the test, and

M = normality of 4-chlorobenzene diazo solution.

A-3 DETERMINATION OF β-NAPHTHOL, G SALT AND R SALT

A-3.1 Outline of the Method

The impurities in Schaeffer's acid (sodium salt) is determined by using descending paper chromatographic technique.

A-3.2 Apparatus

A-3.2.1 *Developing Chamber*

A-3.2.2 *Chromatographic Sprayer*

A-3.2.3 *Micropipette*

A-3.2.4 *UV Lamp*

A-3.3 Reagents

A-3.3.1 *G Salt*, 0·1 percent solution (on 100 percent basis) in 3 N ammonia solution water (1 : 9).

A-3.3.2 *R Salt*, 0·1 percent solution (on 100 percent basis) in 3 N ammonia solution + water (1 : 9).

A-3.3.3 *β-Naphthol*, 0·5 percent solution (on 100 percent basis) in 3 N ammonia solution + water (1 : 9).

A-3.3.4 *Mobile Phase*, 40 percent aqueous solution of calcium chloride.

A-3.3.5 *Schaeffer's Salt*, free from R salt, G salt and β-naphthol.

A-3.3.6 *Spray Reagent*, 0·1 percent aqueous solution of diazo fast red B salt.

A-3.4 Procedure

A-3.4.1 First prepare a standard solution of Schaeffer's salt containing known amounts of R salt, G salt and β-naphthol. Into each of three 100-ml volumetric flasks, weigh accurately 1·0 g of Schaeffer's salt (**A-3.3.5**). Add 8·0, 9·0 and 10·0 ml of 0·1 percent solution of G salt (**A-3.3.1**) in ammonia solution to flask No. 1, 2 and 3 respectively. Dissolve the contents of the flask in ammonia hydroxide solution. Now, there are 3 solutions of 0·8, 0·9 and 1·0 percent G salt content. In a fourth 100 ml flask, weigh accurately 1·0 g of the prepared sample (*see* **A-1.1**), dissolve in ammonium hydroxide solution and dilute to 100 ml with ammonium hydroxide solution.

A-3.4.1.1 Prepare a similar set of three solutions of 0·08, 0·09 and 0·1 percent of R salt and 0·4, 0·45 and 0·5 percent of β-naphthol content with the use of 0·1 percent R salt (**A-3.3.2**) and β-naphthol (**A-3.3.3**) solution in ammonia.

A-3.4.2 Place 10 µl spot of each of the three solutions of G Salt, R Salt, β-naphthol and prepared sample solution (**A-3.4.1**) using micropipette in the same line to a distance of about 40 mm on filter paper (Whatman No. 1 or equivalent). Place the paper in a descending paper chromatographic chamber containing the mobile phase (**A-3.3.4**) and previously saturated with the same reagent. Allow the solvent to run in a descending manner for about 300 mm from the spot. This will take about 7 hours. Take out the paper after 300 mm run and dry the solvent completely and observe under UV light. Spray the paper with spray reagent (**A-3.3.6**) and develop the chromatogram.

A-3.4.2.1 Develop a similar chromatogram with three solutions of R salt and β-naphthol along with prepared sample solution (**A-3.4.1**).

The separated spot characteristic of constituents may be identified by their colour as under:

Contents	*Zone*	*Rf Value*	*Colour*	
			Under UV Light	After Spray
β-naphthol	I	0·16	—	Pink
Schaeffer's salt	II	0·33	Bluish	Pink
G Salt	III	0·62	Bluish	Pink
R Salt	IV	0·72	Bluish	Pink

A-3.5 Report

Report G salt, R salt and β-naphthol as that which is nearest in intensity to the standard. In case the colour intensity does not come in the range of standard spots, repeat the whole procedure using different percentage of G salt, R salt and β-naphthol.

A-4 MATTER INSOLUBLE IN SODIUM CARBONATE SOLUTION

A-4.1 Reagents

A-4.1.1 *Sodium Carbonate Solution*, 5 percent (*m/v*), filtered free from suspended impurities.

A-4.2 Procedure

Weigh about 5·0 g of the material in 500-ml beaker. Add 25 ml each of water and sodium carbonate solution. Warm to 60°C and cool to room temperature.

Filter through sintered glass crucible G3 and wash with hot water till free from alkali. Dry the residue at 100°C to a constant mass.

A-4.3 Calculation

$$\text{Matter insoluble in sodium carbonate solution, percent by mass} = \frac{M_1 - M_2}{M} \times 100$$

where

M_1 = mass in g of sintered crucible with residue,

M_2 = mass in g of empty sintered crucible, and

M = mass in g of sample taken for test.

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